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DEPARTMENT OF PHYSICS 0319 9500 GILMAN DRIVE LA JOLLA, CALIFORNIA 92093-0319 Telephone: (619)-534-2047 FAX: (619)-534-2232 e-mail:rdynes@ucsd.edu

June 27, 2001

Dr. Harold Weinstock, Program Manager Air Force Office of Scientific Research Directorate of Physics & Electronics AFOSR/NE 801 N. Randolph Street, Room 732 Arlington, VA 22203-1977

Dear Harold,

Attached is my final report on my AFOSR/DURIP F49620-99-1-0171, Construction of an Advanced Vacuum Deposition System for Fabrication of Tunneling and Nanostructured Magnetic Devices.

Hope all is well.

Regards.

Robert C. Dynes Professor of Physics Chancellor, UCSD

RCD/cr

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AFOSR/DURIP F49620-99-1-0171

Construction of an Advanced Vacuum Deposition System for Fabrication of Tunneling and Nanostructured Magnetic Devices
Final Report 04/01/99-03/31/01
Submitted June 27, 2001
Professor Robert C. Dynes
University of California, San Diego

The purpose of this project was to build a deposition chamber dedicated to the fabrication of magnetic thin films and thin film structures to study electron transport and interactions in magnetic tunnel junctions and nanostructures. The laboratory of the PI has the capabilities for the deposition of conventional non-magnetic metals and a laser ablation system for the deposition of oxide metals and superconductors, but as the interests have been moving toward the interactions of metals and superconductors with magnetic materials, a system exclusively committed to this end was requested and built. The deposition chamber was designed to be as flexible as possible to incorporate various deposition techniques and a wide range of substrate configurations and temperatures. Thermal resistance and electron beam multiple hearth sources have been incorporated in the original system and provisions for the future addition of at least two sputter guns have been included. Furthermore, as we are interested in depositing materials on to substrates held at a wide range of temperatures, the system affords the possibility of substrate temperatures from 77K to 700K.

The budget for this chamber was \$95,000 and is described below.

| Equipment | <u>Cost</u> | <u>Vendors</u> |
|---------------------------------|-------------|-----------------------|
| Vacuum Chamber/Frame | \$15,000 | MDC |
| Vacuum Pumps | 18,000 | CTI |
| Gate Valve | 5,000 | VAT |
| Hoist/Gantry | 1,500 | McMaster-Carr |
| Electron Beam Source/Controller | 20,000 | MDC |
| 100 A Power Supply | 5,000 | HP |
| Deposition Thickness Monitors | 5,000 | Infinicon |
| Substrate Heater | 1,000 | UCSD SIO Machine Shop |
| Substrate Heater Controller | 2,500 | Eurotherm |
| Pressure Gauge Controller | 2,500 | Granville-Phillips |
| Cold Stage | 5,000 | UCSD SIO Machine Shop |
| Mask Changer | 5,000 | UCSD SIO Machine Shop |
| Motion Feedthroughs | 5,000 | MDC |
| Computer | 1,500 | ComLogic |
| GPIB Controller Card | 1,000 | National Instruments |
| Lab View Software | 2,000 | National Instruments |
| TOTAL | \$95,000 | |

The AFOSR award was for \$75,000 and UCSD internal funds of \$20,000 were committed to meet the anticipated costs.

The chamber and components listed above have been fabricated and assembled in the PI's laboratory. The chamber is shown in Figure 1. All components, controllers and sources have been purchased and are either assembled or are ready to be assembled. The final assembly of some of the components and the substrate holders is not complete as the students and postdoctoral fellows who were responsible for the original design have graduated and moved to new positions. We are awaiting the arrival of a new postdoctoral fellow (arriving July/2001) who will complete the building project. The system has been leak tested and has successfully pumped to the 10^{-10} torr scale. We anticipate the chamber to be in full operation within six months of this report.

We have kept close to the original budget in the construction of this chamber. The over runs will be accommodated by internal UCSD funds. This chamber was designed such that it had the capability of being very flexible to that it could accommodate unanticipated future opportunities. For example, we designed into the chamber the option of retrofitting two or three sputter guns. It is expected that soon after the chamber is fully functional, we will be pursuing this option.

When functioning, it is anticipated that this deposition chamber will greatly enhance the functionality of the PI's laboratory. Currently we are engaged in research in high Tc superconducting electronics, the electronic properties of superconducting/normal metal interfaces, electrical properties in reduced dimension (0d, 1d and 2d) and nanomagnetism. We are capable of fabricating structures as thin as 1nm and lateral dimensions on length scales of a few tens of nanometers. Magnetic structures of this size have particular interest because on this scale effects of superparamagnetism and domain wall energies become particularly acute. We have show ^{1,2} (References 201 and 203] that on this scale, superparamagnetism and the influence of the proximity effect between a ferromagnet and a conventional metal can profoundly affect the magnetic properties of thin magnetic films. From this work it was clear to us that the chamber that is the subject of this project was necessary to pursue the interesting results of our earlier work.

Undergraduates

Taryl Kirk, African American Male, now at IBM Terri Lisman, Female, now a graduate student at University of California, Santa Cruz

Graduates Students

Jeffrey Ostrick, now at TRW in superconductivity group

Post Doctorates

Andrew Katz, now at Arthur Anderson Aviad Frydman, Assistant Professor, Bar Ilan University, Ramat Gan, Israel Olivier Bourgeois, now at Grenoble

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¹ A. Frydman, L.M. Merchant, R.C. Dynes, Proximity Effects in Quench-Condensed Granular Superconductors and Ferromagnets, *Phys. Stat. Sol.* (b), Vol. **218**, p.173

² A. Frydman, TlL. Kirk, R.C. Dynes, SSC, 2/2000 Vol. 114 p. 481



Figure 1. Vacuum Chamber

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Proximity Effects in Quench-Condensed Granular Superconductors and Ferromagnets

A. FRYDMAN (a, b), L. M. MERCHANT (b), and R. C. DYNES (b)

- (a) Department of Physics, Bar Ilan University, Ramat Gan 52900, Israel Tel.: 972-3-5318102, Fax: 972-3-5353298, e-mail: frydman@mail.biu.ac.il
- (b) Department of Physics, University of California, San Diego, La Jolla, CA 92093, USA

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We report on a study of proximity effects in quench-condensed superconducting (Pb) and ferromagnetic (Ni) granular films in which we are able to deposit sequential layers of material and measure transport and magnetoresistance properties in-situ for different degrees of material coverage. We find that adding a normal (non-superconducting or non-ferromagnetic) overlayer to isolated Pb or Ni grains enhances long-range order in the film. We interpret these results as being due to proximity effects where the grains are coupled via the weak order-parameter induced in the overlayer material.

Superconductivity and magnetism are two fundamental phenomena that exhibit internal ordering and can be characterized by a Ginsburg-Landau order-parameter. It seems compelling to try to find analogies between the two. A magnetic analogue to the superconducting proximity effect, in which the magnetization varies smoothly across a ferromagnet/non-magnetic metal interface, has been considered in the past [1 to 4]. However, the experimental realization of this effect is rather challenging since the coherence length of a typical ferromagnet is of the order of a few atomic spacings. In this paper we describe measurements of granular films of Pb and Ni covered by sequential overlayers of different materials enabling control over the coupling between the grains. We find indications for superconducting and magnetic proximity effects in which the overlayers couple between the isolated grains and enhance long-range order.

The samples were prepared using the "quench-condensation" technique, i.e. evaporation on a cryo-cooled ($T=4\,\mathrm{K}$) substrate under UHV conditions within the measurement apparatus as described elsewhere [5]. This method enables one to study the properties of a single sample as a function of the amount of deposited material while keeping the sample at low temperatures and in UHV environment. It is especially suitable for proximity effect experiments since the ultra high vacuum gives rise to barrier-free interfaces between different evaporated materials and the low substrate temperature all but eliminates inter-metallic diffusion. For thin enough films on a passivated substrate, the quench-condensed layer contains discontinuous grains with diameters of a few hundreds of Å and heights of 30 to 70 Å [5, 6]. As more material is deposited, grains begin to coalesce. The average grain size thus increases and inter-grain spacing decreases until, beyond a percolation threshold, the film becomes continuous.

Figure 1a shows resistance versus temperature curves of a quench-condensed Pb film for different deposition steps. Upon adding layers of material the sample resistance

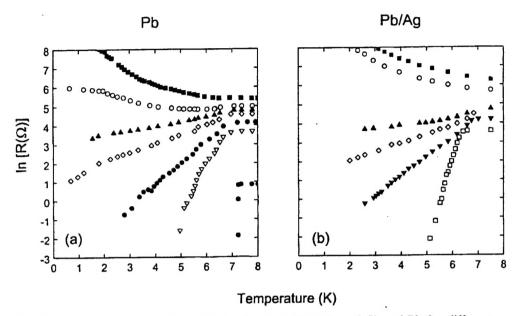


Fig. 1. a) Resistance versus temperature of a quench-condensed film of Pb for different evaporation steps. The nominal thickness are: 60 Å (squares), 61 Å (empty circles), 61.4 Å (up triangles), 61.8 Å (diamonds), 62.7 Å (full circles), 64 Å (down triangles) and 75 Å (circles). b) Similar measurements for a 60 Å thick layer of Pb covered by a layer of Ag with a nominal thickness of 0.5 Å (full squares), 1 Å (circles), 1.5 Å (up triangles), 3 Å (diamonds) and 5 Å (empty squares).

decreases and the film undergoes an insulator to superconductor transition. It is noted that the superconducting transition gets sharper as the normal state resistance is reduced, while the temperature at which the resistance starts dropping rapidly varies only slightly throughout the entire superconductor—insulator transition. This implies that each grain sustains bulk superconductive properties [7]. The insulator—superconductor transition is due to the fact that individual grains couple and increasing areas of superconducting phase-coherence immerge.

Our next step was to prepare isolated Pb grains in the insulating state (such as the top curve in Fig. 1a) and to add overlayers of a normal metal (Ag). Figure 1b depicts the R-T curves of such films. It is seen that the overlayers of Ag cause an insulator-superconductor transition similar to that of adding the superconducting Pb layers. This can be understood by means of the proximity effect. Individual Pb grains are coupled via the weak superconductivity induced in the Ag layer.

The analogous experiment for the magnetic proximity effect is performed in a similar way. Figures 2a to c show the MR curves of sequentially quench-condensed layers of Ni. A crossover from non-hysteretic to hysteretic curves is observed as material is added. Evidently, the thinnest film consists of small grains which are isolated from each other and are superparamagnetic at $T=4\,\mathrm{K}$. Thus, when the field is removed, the thermal energy is large enough to randomize the spin orientation of each grain independently and the MR curve does not exhibit hysteretic behavior. As more material is deposited the grains coalesce, the effective magnetic domain sizes become larger, the superparamagnetic blocking temperature rises above $4\,\mathrm{K}$ and the film exhibits ferromagnetic behavior at $T=4\,\mathrm{K}$. Hence, the curves become hysteretic. Indeed, the tem-

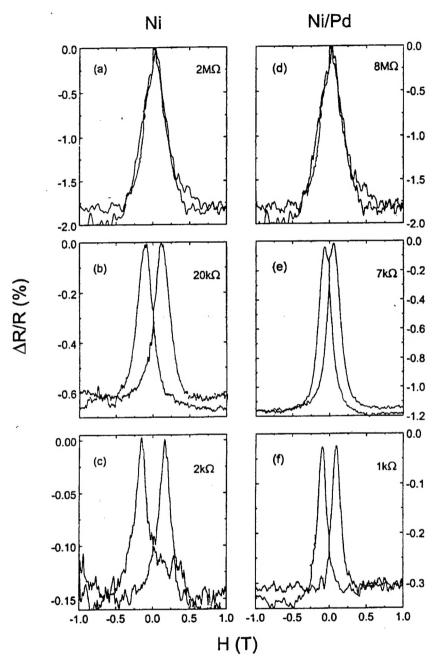


Fig. 2. Left: Magnetoresistance curves at T = 4.2 K of a quench-condensed Ni film for different steps of the evaporation. The field was swept from -1 T to 1 T and back. The nominal film thicknesses were 20 Å (a), 21 Å (b) and 25 Å (c). Right: Similar measurement of a film which consists of a 20 Å film of Ni covered by 0 Å (d), 2 Å (e) and 6 Å (f) layers of Pd

perature dependence of these MR curves is consistent with well-known relations for the superparamagnetic transition [8].

Having observed the above effect of adding magnetic material to a granular magnetic film, we proceeded to study the effects of adding non-magnetic materials to superpara-

magnetic grains. We prepared films of isolated Ni grains (20 Å nominal thickness), which showed no hysteresis in the MR curve, and we added overlayers of Pd, Ti, Ag or Cu in-situ. The MR curves of a sample in which overlayers of Pd were added to the 20 Å Ni film are shown in Figs. 2d to f. Despite the fact that Pd is non-ferromagnetic, its presence gives rise to a hysteresis in the MR. Such a hysteresis is indicative of coupling between magnetic grains which were originally isolated. Similar behavior was observed for the other overlayer materials, however the coupling strength of the medium (measured by the coercive field, H_c , at which the MR reaches a maximum) was correlated with the material magnetic susceptibility. Pd, which is a strong paramagnet ($\chi \approx 1.1 \times 10^{-6}$ emu/g Oe) has an effect that is nearly as strong as that of adding Ni itself. Ti, a weaker paramagnet ($\chi \approx 1.5 \times 10^{-6}$ emu/g Oe), has a significant but smaller coupling coefficient. Diamagnetic materials, such as Ag or Cu, also couple between the Ni grains, but the influence of diamagnetic overlayers is much weaker than that of paramagnets [5].

The correlation between the coupling strength and the magnetic susceptibility of the intermediate material, supports the hypotheses that the magnetic coupling through the normal medium bears the nature of a proximity effect rather then a Coulomb effect such as the RKKY mechanism. The fact that we observe a strong correlation of the degree of hysteresis with magnetic susceptibility implies that the relevant coupling mechanism is one in which a magnetic moment is induced in the intermediate medium, which, in turn, couples the magnetic grains. We note the resemblance between the experiments in superconducting and ferromagnetic grains. In both cases we start with a system that has no long-range order and by adding overlayers of material (which has no internal order), we cause a crossover to an ordered system.

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